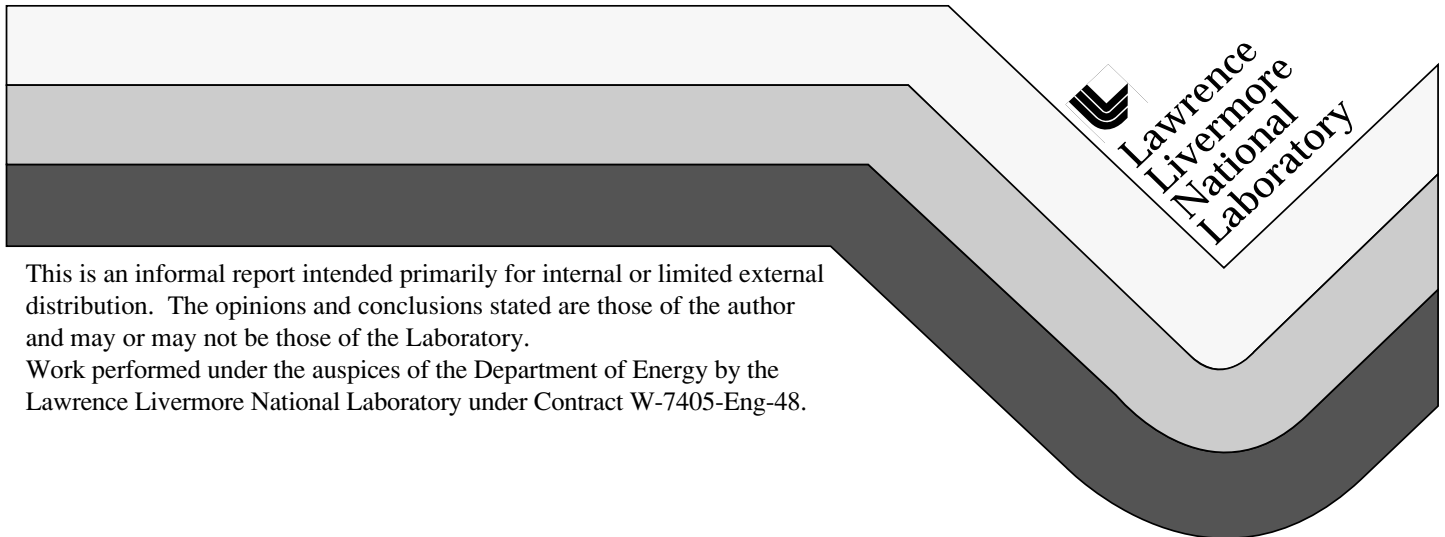


**DISTRIBUTION OF SOLUBLE AND PRECIPITATED IRON AND CHROMIUM PRODUCTS
GENERATED BY ANODIC DISSOLUTION OF 316L STAINLESS STEEL AND ALLOY C-22:
FINAL REPORT**

Joe Farmer,
Dave Silberman
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Ken King
Steve Gordon
Larry Logotetta

August 1999



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CONTENTS

	Page
1. CORROSION EXPERIMENTS.....	1
2. ANALYSES OF CHROMIUM IN SOLUTIONS FROM CORROSION EXPERIMENTS	1
2.1 SAMPLE PREPARATION.....	1
2.2 CHROMIUM VI ANALYSIS	2
2.3 TOTAL CHROMIUM ANALYSIS.....	2
2.4 RESULTS FOR CHROMIUM.....	2
3. ANALYSES OF IRON IN SOLUTIONS FROM CORROSION EXPERIMENTS.....	3
3.1 SAMPLE PREPARATION.....	3
3.2 FE(II) ANALYSIS	3
3.3 TOTAL FE ANALYSIS.....	4
3.4 RESULTS FOR FE ANALYSES	4
4. SUMMARY	5
5. REFERENCES.....	5

TABLES

	Page
Table 1. Summary of Conditions for Experiments with Artificial Crevices (at 20°C).....	6
Table 2. Chromium Analyses from Experiments with Artificial Crevices (at 20°C).....	7
Table 3. Iron Analyses from Experiments with Artificial Crevices (at 20°C).....	8
Table 4. Summary of Conditions for Cyclic Polarization Measurements.....	9
Table 5. Chromium Analyses for Cyclic Polarization Measurements	10
Table 6. Iron Analyses for Cyclic Polarization Measurements.....	11

1. CORROSION EXPERIMENTS

Thus far, electrolytes with dissolved and precipitated metal have been collected from two generic types of corrosion experiments: crevice corrosion experiments described in detail in Activity Plan E-20-81 Rev. 2 found in scientific notebook (SN) #00419 (consistent with Activity Plan E-20-89 found in SN #00420); and cyclic polarization experiments described in detail in Activity Plan E-20-43/44 found in SN #00424 and SN #00436. Crevice experiments are summarized in Table 1, and cyclic polarization measurements are summarized in Table 4. These electrolytes have been analyzed to determine the quantities of dissolved and precipitated metal and the oxidation states of dissolved species.

2. ANALYSES OF CHROMIUM IN SOLUTIONS FROM CORROSION EXPERIMENTS

2.1 SAMPLE PREPARATION

The oxidation state of chromium in each of 28 electrolyte samples from corrosion experiments was determined. The plan was to determine the total Cr and Cr(VI) concentrations, thereby making it possible to estimate the Cr(III) concentration by difference. In this study, samples were stored prior to analysis for approximately one week without refrigeration.

Thirteen samples contained either a brown-orange or a white precipitate. The brown-orange precipitate was thought to be precipitated iron and chromium. Each sample bottle was weighed and then shaken vigorously to uniformly suspend the precipitate. An aliquot was rapidly poured into and filtered through a 0.2- μ m polyethersulfone 50- μ m membrane filtration unit (Nalgene 165-0020). The sample bottle was reweighed to obtain the aliquot weight taken. The density of the filtrate was measured, and the volume of the aliquot taken for filtration was determined. The filtrate was transferred to 250-ml polyethylene bottle. This was repeated for each of the 13 samples.

The remaining precipitate on the filter was washed three times (3X) with portions of distilled water to remove any residual solution, and the washings were discarded. The precipitate was extracted by allowing 5 ml of 6 M redistilled-grade HCl to sit on the sample and dissolve it. The precipitate was washed with 0.1 N H₂SO₄ and collected into the tared receiving flask, which is the bottom portion of the filtration unit. The contents of the receiving flask were poured back onto the precipitate and allowed to extract it again. The precipitate was washed three times (3X) with small portions of distilled water, and the total volume of the extracted precipitate was stored in the receiving flask. The receiving flask was reweighed to obtain the extract weight captured. The density of the extract was measured, and the volume of the extract obtained was determined. This was repeated for each of the 13 samples.

2.2 CHROMIUM VI ANALYSIS

The samples that had no precipitate were analyzed as received. The filtrates, extracts, and unfiltered solutions were analyzed for Cr(VI) using Environmental Protection Agency (EPA) method 7196A.

2.3 TOTAL CHROMIUM ANALYSIS

To obtain total chromium, any Cr(III) was first be oxidized to Cr(VI) and then analyzed by EPA method 7196A. Aliquots of the filtrates, extracts, and unfiltered solutions were oxidized using the procedure outlined in Method 3500-Cr B Colorimetric Method in the 20th Edition of *Standard Methods for Water and Wastewater*. Depending on chromium concentration, aliquots ranging from 0.1 ml to 10 ml were taken. As an aliquot was acidified, a drop of saturated potassium permanganate was added, and the sample was boiled. The excess potassium permanganate was reduced with 1 to 2 ml of 0.5% (w/v) sodium azide solution. As before, the samples were then analyzed for Cr(VI). If the total Cr analysis was equal to the Cr(VI) analysis without the oxidation step, it was assumed that no Cr(III) was present.

The samples were analyzed using a Cary 2300 UV-VIS spectrophotometer at 540 nm. The instrument was zeroed with distilled water in 1-cm quartz cuvettes. A 1.0 µg/ml Cr(VI) solution had an absorbance reading of 0.8. The standard curve generated is linear to 2.0 µg/ml Cr(VI). The data are reported as µg Cr/ml of original shaken aliquot.

2.4 RESULTS FOR CHROMIUM

Chromium analyses for the crevice experiments and cyclic polarization measurements are found in Table 2 and Table 5, respectively. Additional details have been published elsewhere (Farmer et al. 1999). In mild electrolytes and at low applied potential, little or no dissolved chromium could be detected (denoted ND). However, in some cases precipitates were formed. At near neutral pH and at applied potentials above the threshold potential for localized breakdown of the passive film, virtually all of the dissolved chromium appears to be in the hexavalent oxidation state (Cr(VI)). In acidic environments, such as crevice solutions formed during the crevice corrosion of 316L and C-22 samples in 4 M NaCl, virtually all of the dissolved chromium appears to be in the trivalent oxidation state (Cr(III)). These general observations appear to be consistent with the Pourbaix diagram for chromium (Pourbaix 1974), pp. 256—271. At high pH and high anodic polarization (pH~8 and 800 mV vs. SHE), the predominate species is believed to be the soluble chromate anion (CrO_4^{2-}). At the same pH, but lower polarization (pH~8 and 0 mV vs. SHE), the predominate species are believed to be precipitates such as trivalent $\text{Cr}(\text{OH})_3 \cdot n(\text{H}_2\text{O})$ and hexavalent Cr_2O_3 . In acidified environments such as those found in crevices (pH<3), soluble Cr^{3+} is expected to form over a wide range of potential extending from -400 mV vs. SHE to

approximately 1200 mV vs. SHE. Again, this is consistent with the observations from the creviced samples. In earlier studies by the principal investigator, it has been found that low-level chromium contamination in groundwater is usually in the hexavalent oxidation state (Farmer et al. 1996).

3. ANALYSES OF IRON IN SOLUTIONS FROM CORROSION EXPERIMENTS

3.1 SAMPLE PREPARATION

An analytical request was made to determine the oxidation state of iron in each of 28 water samples. The plan was to determine both total Fe and Fe^{+2} giving Fe^{+3} by difference. Because the samples were already a week old before they reached the principal investigator, they were not refrigerated.

Thirteen samples contained either a brown-orange or a white precipitate. The brown-orange precipitate was thought to be precipitated iron and chromium. Each sample bottle was weighed and then shaken vigorously to uniformly suspend the precipitate. An aliquot was rapidly poured into and filtered through a 0.2- μm polyethersulfone 50- μm membrane filtration unit (Nalgene 165-0020). The sample bottle was reweighed to obtain the aliquot weight taken. The density of the filtrate was measured, and the volume of the aliquot taken for filtration was determined. The filtrate was transferred to a 250-ml polyethylene bottle. This was repeated for each of the 13 samples.

The remaining precipitate on the filter was washed three times (3X) with portions of distilled water to remove any residual solution, and the washings were discarded. The precipitate was extracted by allowing 5 ml of 6 M redistilled-grade HCl to sit on the sample and dissolve it. The precipitate was washed with 0.1 N H_2SO_4 and collected into the tared receiving flask, which is the bottom portion of the filtration unit. The contents of the receiving flask were poured back onto the precipitate and allowed to extract it again. The precipitate was washed three times (3X) with small portions of distilled water, and the total volume of the extracted precipitate was stored in the receiving flask. The receiving flask was reweighed to obtain the extract weight captured. The density of the extract was measured, and the volume of the extract obtained was determined. This was repeated for each of the 13 samples.

3.2 FE(II) ANALYSIS

The samples that had no precipitate were analyzed as received. The filtrates, extracts, and unfiltered solutions were analyzed for Fe^{+2} and total Fe using method 3500-Fe B Phenanthroline Method (pages 76-78) in the 20th Edition of *Standard Methods for the Examination of Water and Wastewater*.

3.3 TOTAL FE ANALYSIS

depending on iron concentration, aliquots of the filtrates, extracts, and unfiltered solutions ranging from 0.1 ml to 10 ml were taken and acidified with 2 ml of concentrated HCl. To obtain total Fe, 1 ml of 10% hydroxylamine hydrochloride was added followed by 10 ml of ammonium acetate buffer and 4 ml of phenanthroline solution. The samples were diluted to exactly 100 ml and analyzed for total Fe^{+2} as before. If the total Fe analysis with the reduction step was equal to the Fe^{+2} analysis without the reduction step, it was assumed that Fe^{+3} was not present.

The samples were analyzed using a Cary 2300 UV-VIS spectrophotometer at 510 nm. The instrument was zeroed with distilled water in 1-cm quartz cuvettes. A $2.0 \mu\text{g/mL Fe}^{+2}$ solution had an absorbance reading of 0.4. The standard curve generated is linear to $10.0 \text{ (g Fe}^{+2}\text{ / ml)}$. The data are reported as $\mu\text{g Fe/ml}$ of original shaken aliquot.

The quantity of Fe^{+3} can be determined by calculating the difference between the total Fe and the Fe^{+2} .

3.4 RESULTS FOR FE ANALYSES

Iron analyses for the iron measurements are found in Table 3 and 6, respectively. In most instances, virtually all of the iron was precipitated. In the case of crevice experiment 2 Q (316L in saturated KCl), a high concentration of Fe(II) was detected in the actual crevice solution. Approximately 90% of the crevice solution was in the +2 oxidation state, with the remaining 10% in the +3 oxidation state. In this case, all Fe found outside of the crevice mouth was in the form of a precipitate. In the case of crevice experiment 25 Q (C-22 in SCW with the addition of 4 moles per liter of NaCl) and crevice experiment 26 Q (C-22 in SCW), the dissolved Fe was in a relatively high oxidation state (Fe(III)). In this case, the measured pH values at the end of the experiment were 7.08 and 8.50, respectively (Table 1). In general, dissolved Fe measured during the crevice experiments appeared to be Fe(II) in acidic media and Fe(III) in near-neutral and alkaline solutions (Table 3).

In the case of cyclic polarization measurements, the dissolved Fe measured at the end of some cyclic polarization measurements with C-22 appeared to be in the Fe(III) state. This is probably due to the high electrochemical potential at which these species were generated during the potential scan. Note that the reversal potential was approximately 1200 mV vs. Ag/AgCl during these scans (Table 6).

These results are also consistent with the corresponding Pourbaix diagrams. For example, in acidic media (pH 0 to 1), Fe^{+2} would be expected to form at relatively low potential, with conversion to Fe^{+3} at high applied potential (greater than about 700 mV vs. SHE). In neutral to alkaline pH, precipitates of Fe(OH)_2 , Fe(OH)_3 , Fe_2O_3 , or Fe_3O_4 would be expected, with some hydrolyzed species of Fe^{+3} possible.

4. SUMMARY

At near neutral pH and at applied potentials above the threshold potential for localized breakdown of the passive film, virtually all of the dissolved chromium appeared to be in the hexavalent oxidation state (Cr(VI)). In acidic environments, such as crevice solutions formed during the crevice corrosion of 316L and C-22 samples in 4 M NaCl, virtually all of the dissolved chromium appeared to be in the trivalent oxidation state (Cr(III)). These general observations appear to be consistent with the Pourbaix diagram for chromium (Pourbaix 1974), pp. 307–321. At high pH and high anodic polarization (pH~8 and 800 mV vs. SHE), the predominate species is believed to be the soluble chromate anion (CrO_4^{2-}). At the same pH, but lower polarization (pH~8 and 0 mV vs. SHE), the predominate species are believed to be precipitates such as trivalent $\text{Cr}(\text{OH})_3 \cdot n(\text{H}_2\text{O})$ and hexavalent Cr_2O_3 . In acidified environments such as those found in crevices (pH<3), soluble Cr^{3+} is expected to form over a wide range of potential extending from –400 mV vs. SHE to approximately 1200 mV vs. SHE. Again, this is consistent with the observations from the creviced samples. In earlier studies by the principal investigator, it has been found that low-level chromium contamination in ground water is usually in the hexavalent oxidation state (Farmer et al. 1996).

In general, dissolved iron measured during the crevice experiments appears to be Fe(II) in acidic media and Fe(III) in near-neutral and alkaline solutions (Table 3). In the case of cyclic polarization measurements, the dissolved iron measured at the end of some cyclic polarization measurements with C-22 appeared to be in the Fe(III) state. This is probably due to the high electrochemical potential at which these species were generated during the potential scan. Note that the reversal potential was approximately 1200 mV vs. Ag/AgCl during these scans (Table 6). These results are also consistent with the corresponding Pourbaix diagrams. For example, in acidic media (pH 0 to 1), Fe^{+2} would be expected to form at relatively low potential, with conversion to Fe^{+3} at high applied potential (greater than about 700 mV vs. SHE). In neutral to alkaline pH, precipitates of $\text{Fe}(\text{OH})_2$, $\text{Fe}(\text{OH})_3$, Fe_2O_3 , or Fe_3O_4 would be expected, with some hydrolyzed species of Fe^{+3} possible.

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Table 1. Summary of Conditions for Experiments with Artificial Crevices (at 20°C) [DTN LL990802205924.086]

Expt.	ID #	Alloy	Test Medium	E _{corr} —High mV vs. Ag/AgCl	E _{corr} —Low mV vs. Ag/AgCl	E _{applied} mV vs. Ag/AgCl	Duration — hours	Electrolyte pH—Start	Electrolyte pH—Finish	Crevice pH—Start	Crevice pH—Finish
Crevice	02 Q	316LN	Satd. KCl— Reservoir	–237.60	–242.41	800	37	7.09	6.18	7.22	1.24
Crevice	02 Q	316LN	Satd. KCl— Crevice Purge	–237.60	–242.41	800	37	7.09	6.18	7.22	1.24
Crevice	11 Q	316L	4 M NaCl	–117.21	–133.34	800	3	8.02	10.18	7.95	0.97
Crevice	12 Q	316L	4 M NaCl	–146.54	–156.31	800	39	8.30	8.42	7.42	1.50
Crevice	13 Q	316L	4 M NaCl	–127.65	–189.82	400	20	6.43	8.89	7.96	1.24
Crevice	14 Q	316L	4 M NaCl	–146.23	–135.63	200	20	6.91	8.59	7.69	1.09
Crevice	15 Q	316L	4 M NaCl + SCW								
Crevice	21 Q	C-22	4 M NaCl	–107.70	–150.50	800	23	8.04	6.71	8.21	6.97
Crevice	22 Q	C-22	4 M NaCl—Reservoir	–97.50	–163.20	400	66	8.14	6.98	8.50	4.69
Crevice	22 Q	C-22	4 M NaCl—Cell	–97.50	–163.20	400	66	8.14	6.98	8.50	4.69
Crevice	23 Q	C-22	4 M NaCl	–89.60	–118.50	1100	26	6.98	8.99	4.69	0.38
Crevice	24 Q	C-22	4 M NaCl + SCW	–132.50	–143.20	1100	5	8.04	8.04	8.04	6.77
Crevice	25 Q	C-22	4 M NaCl + SCW	–75.53	–1.96	1100	5	8.25	8.24	8.21	7.08
Crevice	26 Q	C-22	SCW	–120.50	–133.30	0 to 800	70	8.25	8.55	8.23	8.50
Crevice	27 Q	C-22	4 M NaCl	–64.70	–97.80	800	116	8.26	8.44	7.24	–0.05

Table 2. Chromium Analyses from Experiments with Artificial Crevices (at 20°C) [DTN LL990802205924.086]

Expt.	ID #	Alloy	Test Medium	Total Dissolved Cr (µg/ml)	Dissolved Cr(VI) (µg/ml)	Dissolved Cr(III) (µg/ml)	Dissolved Cr MDL (µg/ml)	Cr Precipitate (µg/ml)	Precipitated Cr MDL (µg/ml)
Crevice	02 Q	316LN	Satd. KCl—Reservoir	ND	ND	ND	0.1	7.5	0.1
Crevice	02 Q	316LN	Satd. KCl—Crevice	170	ND	170	10	no analysis	no analysis
Crevice	11 Q	316L	4 M NaCl	ND	ND	ND	0.1	ND	0.1
Crevice	12 Q	316L	4 M NaCl	ND	ND	ND	0.1	12.0	0.1
Crevice	13 Q	316L	4 M NaCl	ND	ND	ND	0.1	3.6	0.1
Crevice	14 Q	316L	4 M NaCl	ND	ND	ND	0.1	20.0	0.1
Crevice	15 Q	316L	4 M NaCl + SCW	ND	ND	ND	0.1	ND	0.1
Crevice	21 Q	C-22	4 M NaCl	ND	ND	ND	0.1	ND	0.1
Crevice	22 Q	C-22	4 M NaCl—Reservoir	ND	ND	ND	0.1	2.0	0.1
Crevice	22 Q	C-22	4 M NaCl—Cell	ND	ND	ND	0.1	3.3	0.1
Crevice	23 Q	C-22	4 M NaCl	0.9	0.8	0.1	0.1	6.6	0.1
Crevice	24 Q	C-22	4 M NaCl + SCW	no analysis	no analysis	no analysis	no analysis	no analysis	no analysis
Crevice	25 Q	C-22	4 M NaCl + SCW	0.8	0.8	ND	0.1	ND	0.1
Crevice	26 Q	C-22	SCW	2.5	2.3	0.2	0.1	2.1	0.1
Crevice	27 Q	C-22	4 M NaCl	no analysis	no analysis	no analysis	no analysis	no analysis	no analysis

Table 3. Iron Analyses from Experiments with Artificial Crevices (at 20°C) [DTN LL990802205924.086]

Expt.	ID #	Alloy	Test Medium	Total Dissolved Fe (µg/ml)	Dissolved Fe(III) (µg/ml)	Dissolved Fe(II) (µg/ml)	Dissolved Fe MDL (µg/ml)	Fe Precipitate (µg/ml)	Precipitated Fe MDL (µg/ml)
Crevice	02 Q	316LN	Satd. KCl—Reservoir	ND	ND	ND	0.3	32	4
Crevice	02 Q	316LN	Satd. KCl—Crevice	709	66	643	30	no analysis	no analysis
Crevice	11 Q	316L	4 M NaCl	ND	ND	ND	0.3	ND	3
Crevice	12 Q	316L	4 M NaCl	ND	ND	ND	0.3	48	4
Crevice	13 Q	316L	4 M NaCl	ND	ND	ND	0.3	18	4
Crevice	14 Q	316L	4 M NaCl	ND	ND	ND	0.3	131-148	4
Crevice	15 Q	316L	4 M NaCl + SCW	ND	ND	ND	0.3	ND	3
Crevice	21 Q	C-22	4 M NaCl	ND	ND	ND	0.3	8	3
Crevice	22 Q	C-22	4 M NaCl—Reservoir	ND	ND	ND	0.3	7	4
Crevice	22 Q	C-22	4 M NaCl—Cell	ND	ND	ND	0.3	13	4
Crevice	23 Q	C-22	4 M NaCl	0.8	0.8	ND	0.3	14	4
Crevice	24 Q	C-22	4 M NaCl + SCW	no analysis	no analysis	no analysis	no analysis	no analysis	no analysis
Crevice	25 Q	C-22	4 M NaCl + SCW	0.8	0.8	ND	0.3	ND	3
Crevice	26 Q	C-22	SCW	1.4	1.4	ND	0.3	ND	3
Crevice	27 Q	C-22	4 M NaCl	no analysis	no analysis	no analysis	no analysis	no analysis	no analysis

Table 4. Summary of Conditions for Cyclic Polarization Measurements [DTN LL990802205924.086]

Expt.	ID #	Alloy	Test Medium	Temp. (°C)	E _{corr} —High mV vs. Ag/AgCl	E _{reversal} mV vs. Ag/AgCl	Scan Rate mV/sec	Electrolyte pH—Start	Electrolyte pH—Finish
CP	DEA 025 031899	C-22	SDW	30	–55	1200	0.17	8.61	8.74
CP	DEA 026 031999	C-22	SDW	60	–137	1200	0.17	8.46	9.52
CP	DEA 027 032099	C-22	SDW	90	–191	1200	0.17	8.61	10.12
CP	DEA 024 031899	C-22	SDW	90	–162	1190	0.17	8.38	9.61
CP	DEA 028 032299	C-22	SAW				0.17	2.77	2.95
CP	DEA 029 032299	C-22	SAW	90	–171	1200	0.17	2.77	2.94
CP	DEA 031 032499	C-22	SAW	90	–150	1200	0.17	2.79	2.87
CP	PEA 001 032399	316L	SCW	60	–185	1200	0.17	8.14	9.46
CP	PEA 002 032499	316L	SCW	90	–263	1200	0.17	8.14	9.46
Blank	None	None	4 M NaCl	20	None	None	None		
Blank	None	None	SDW	20	None	None	None		
Blank	None	None	SCW	20	None	None	None		
Blank	None	None	SAW	20	None	None	None		

Table 5. Chromium Analyses for Cyclic Polarization Measurements [DTN LL990802205924.086]

Expt.	ID #	Alloy	Test Medium	Temp. (°C)	Total Unfiltered Cr (µg/ml)	Unfiltered Cr(VI) (µg/ml)	Unfiltered Cr(III) (µg/ml)	Unfiltered Cr MDL (µg/ml)
CP	DEA 025 031899	C-22	SDW	30	ND	0.1	no estimate	0.1
CP	DEA 026 031999	C-22	SDW	60	0.3	0.3	0	0.1
CP	DEA 027 032099	C-22	SDW	90	0.2	0.3	no estimate	0.1
CP	DEA 024 031899	C-22	SDW	90	0.2	0.2	0	0.1
CP	DEA 028 032299	C-22	SAW		7.9	6.1	1.8	0.2
CP	DEA 029 032299	C-22	SAW	90	11.0	8.9	2.1	0.2
CP	DEA 031 032499	C-22	SAW	90	8.4	5.8	2.6	0.2
CP	PEA 001 032399	316L	SCW	60	ND	ND	ND	0.1
CP	PEA 002 032499	316L	SCW	90	ND	ND	ND	0.1
Blank	None	None	4 M NaCl	20	ND	ND	ND	0.1
Blank	None	None	SDW	20	ND	ND	ND	0.1
Blank	None	None	SCW	20	ND	ND	ND	0.1
Blank	None	None	SAW	20	ND	ND	ND	0.1

Table 6. Iron Analyses for Cyclic Polarization Measurements [DTN LL990802205924.086]

Expt.	ID #	Alloy	Test Medium	Temp. (°C)	Total Unfiltered Fe (µg/ml)	Unfiltered Fe(III) (µg/ml)	Unfiltered Fe(II) (µg/ml)	Unfiltered Fe MDL (µg/ml)
CP	DEA 025 031899	C-22	SDW	30				
CP	DEA 026 031999	C-22	SDW	60	ND	ND	0.3	
CP	DEA 027 032099	C-22	SDW	90	ND	ND	0.3	
CP	DEA 024 031899	C-22	SDW	90	ND	ND	0.3	
CP	DEA 028 032299	C-22	SAW		0.8	0.5	0.3	0.3
CP	DEA 029 032299	C-22	SAW	90	0.6	0.6	ND	0.3
CP	DEA 031 032499	C-22	SAW	90	1.2	0.8	0.4	0.3
CP	PEA 001 032399	316L	SCW	60	0.3	0.3	ND	0.3
CP	PEA 002 032499	316L	SCW	90	ND	ND	ND	0.3
Blank	None	None	4 M NaCl	20	ND	ND	ND	0.3
Blank	None	None	SDW	20	ND	ND	ND	0.3
Blank	None	None	SCW	20	ND	ND	ND	0.3
Blank	None	None	SAW	20	ND	ND	ND	0.3